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# **Evaluating Iron-Impregnated Paper Strips for Assessing Available Soil Phosphorus**

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#### **ABSTRACT**

Iron (Fe)-impregnated filter paper strips (Pi) have been proposed as a method for measuring available soil phosphorus (P). A well-defined Pi method has not yet been developed and Pi strips are often prepared with different filter papers and procedures. A study aimed at arriving at a consistent Pi method is thus needed. Four types of Pi strips, prepared with the two most widely used papers, Whatman No. 50 and 541, following a procedure that incorporates improvements both proposed in the literature and made in our laboratory, were evaluated for

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P extraction capacity and error. Two of the best strips. which are significantly different in P extraction capacity, along with the Mehlich 1 (0.05M HCl and 0.0125M  $H_2SO_4$ ) and the Olsen method (0.5M NaHCO<sub>4</sub>, pH 8.5) were further evaluated in a greenhouse experiment involving eight soils planted with corn (Zea mays L.). Results indicated that strips prepared with both Whatman No. 50 and 541 were appropriate for P extractions as long as strips were washed with deionized water after treatment with ammonium hydroxide (NH,OH). At room temperatures the strips probably contain both hydrous Fe hydroxides and oxides in both crystalline and amorphous forms. Pi P was well correlated with Olsen P and P uptake in all soils, indicating that Pi is generally applicable in diverse soils. No obvious advantage was found for the Pi with respect to the Olsen method. Both the Pi and the Olsen method were better extractants with respect to the Mehlich 1, which was ineffective for extracting P in calcareous soils. Extractable P by Mehlich 1, Olsen, and Pi all correlated highly with accumulated plant available P estimated by eight sequential crops in the greenhouse. However, none of the methods could account for all the variation in plant P removal.

#### INTRODUCTION

Accurate estimates of available soil P are often needed for identifying P deficiencies and making reliable recommendations, and sometimes, for detecting soil P excess. Such estimates demand

reliable testing methods. An ideal method is expected not only to discriminate between deficiency and sufficiency of soil P, but also to estimate a proportionate fraction of the available P pool from different soils. In addition, the procedure should work with reasonable accuracy and speed (Kamprath and Watson, 1980).

All existing methods meet one or more of these criteria, but fail in others. For example, destructive chemical extractants remove both available and non-mobile P during extraction (Menon et al., 1989a). Estimates of P by such methods do not properly reflect plant-available P pools in many soils (Fixen and Grove, 1990). For this reason, the non-destructive anion exchange resin method (Amer et al., 1955) has been adopted as an alternative method. Resin P correlated well with plant P uptake in most soils (Bowman et al., 1978; Olsen and Sommers, 1982), but analytical difficulties with the resin method prevent its use in many soil testing laboratories (van der Zee et al., 1987; Menon et al., 1989a; Lin et al., 1991).

The Pi test (van der Zee et al., 1987; Menon et al., 1989a) was proposed as a potential P extraction procedure for measuring available soil P. Pi strips are prepared with filter papers coated with high P-affinity Fe compounds. Similar to the anion exchange resin method, Pi strips do not react with soil components during extraction. Instead, Fe on the strips competes with soil for P in solution. The affinity between Pi strips and P is higher than that between resin and solution P. Analytical difficulties are fewer with Pi extraction than with resin extraction because separation of Pi strips and soil is easier than separation of resin and soil. Therefore, the Pi is considered to have the advantages of the resin method, but is more feasible for measuring

available P in diverse soils amended with different P materials (Menon et al., 1989a; Lin et al., 1991; Sharpley, 1991; Kuo and Jellum, 1994).

In several studies, Pi strips were prepared with different types of filter paper following a variety of preparatory procedures. Various combinations of filter papers and procedures often produce strips of distinct qualities that give different extractable P results. One strip (2 cm x 10 cm) prepared with the same paper (S & S 602) was reported to contain from 79 (Lin et al., 1991) to 120 µmol Fe (van der Zee et al., 1987). Strips prepared with the same Whatman No. 541 paper following the same procedure (Menon et al., 1989a) but in two different laboratories gave significantly different extractable P results for the same samples (Bramley and Roe, 1993). Such inconsistencies are undesirable for inter-laboratory data comparisons. Further evaluations of various strips are necessary to improve the Pi method.

Pi P and P extracted by other methods have been compared in a few studies using strips prepared from various paper and preparation combinations (Menon et al., 1989a; 1989b; Lin et al., 1991; Kuo and Jellum, 1994). A study using Pi strips of the same quality on a wide range of soils will help clarify the adaptability of the Pi method with respect to other existing methods. In addition, the extraction process of Pi strips better mimics the mode of P uptake by plant roots when compared to chemical extractants (van der Zee et al., 1987; Menon et al., 1989c; Lin et al., 1991; Bramley and Roe, 1993). Thus, Pi P is expected to give better estimates of plant available P in soils than extractable P by chemical methods. However, such differences in predictability of plant available P between the Pi and chemical methods need to be further explored.

The objectives of this study were to evaluate extraction capacity and error of strips prepared from two types of filter paper, following two procedures, and to compare the performance of the Pi method with that of two chemical extractants.

#### **MATERIALS AND METHODS**

#### **Preparation of Pi Strips**

Pi strips were prepared with two types of filter paper, Whatman No. 50 (15 cm in diameter, hardened, slow speed), and Whatman No. 541 (15 cm in diameter, hardened, ashless, fast speed), and by the following procedures:

Procedure A: Filter papers were immersed in 0.4M ferric chloride (FeCl<sub>3</sub>) solution for 2 hours (Lin et al., 1991; Sharpley et al., 1994). The papers were taken out and drained of excess FeCl<sub>3</sub>, hung with clothespins and air-dried over-night at room temperature (23-25°C). FeCl<sub>3</sub>-treated papers were immersed in 2.7M ammonium hydroxide solution (NH<sub>4</sub>OH) for 1 minute for even Fe deposition (Sharpley et al., 1994). Papers were then hung to air-dry overnight and cut into 2 cm x 10 cm strips.

Procedure B: Same as in procedure A except that NH<sub>4</sub>OH treated papers were washed with deionized water to remove impurities and unattached Fe after they were dried (Lin et al., 1991; Bramley and Roe, 1993; Kuo and Jellum, 1994). Washing was carried out by directing a jet of deionized water towards a strip held by a pair of tweezers.

The above preparation procedures resulted in four types of Pi strips: (1) Whatman No. 50, washed, (2) Whatman No. 50, unwashed, (3) Whatman No. 541, washed, and (4) Whatman No. 541, unwashed.

#### X-ray Diffraction (XRD) Analysis of Fe

Iron on the strips and that obtained from direct mixing of FeCl<sub>3</sub> with NH<sub>4</sub>OH was analyzed using a Philips XRG-3100 X-ray diffractometer employing a cobalt tube operated at 40 kV and 25 mA. Strips were mounted on molybdenum plates with double-sided tape and scanned from 14 to 80 degrees 20. Precipitate from direct reaction of FeCl<sub>3</sub> and NH<sub>4</sub>OH for 1 minute was filtered and washed five times with deionized water and then air-dried. One-half of the air-dried precipitate was further dried at 105-110°C overnight. Samples of both air-dried and heated precipitate were filled into zero-background quartz mounts and scanned from 4 to 50 degrees 20.

#### Phosphorus Sorption Capacity and Fe Content of Pi Strips

To determine the P sorption capacity of the Pi strips, 0, 100, 200, 300, 400, 500, 600, 800 μg of P as Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O was added in 4 replicates into 40 ml deionized water in 118-ml, 10-cm high, screw-capped glass jars. One Pi strip was added to each jar. Jars were then capped tightly and shaken on a mechanical shaker at 120 rpm for 16 hours (45 minutes every hr). After shaking, strips were washed with deionized water and air dried (25±2°C). The strips were then placed into a separate set of glass jars. Forty ml of 0.2M sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) were added to each jar, which was then shaken for 2 hrs. This caused a complete dissolution of both P and Fe associated with the strips (van der Zee et al., 1987). Both P recovered by the strips and P left in solution were determined by the method of Murphy and Riley (1962). Iron desorbed from each Pi strip was determined by the Perkin-Elmer Model 3030 Atomic Absorption Spectrophotometer.

#### Pi P Extraction Procedure in Soils

One gram (0.84-mm, dry basis) soil was weighed into a 118-ml, screw-capped glass jar. Forty ml of 0.01M CaCl<sub>2</sub> was then added to each jar as a background electrolyte. One Pi strip was placed inside a nylon bag (2.5 cm x 11 cm), which was added to each jar. Jars were then shaken for 16 h. Desorption and measurement of P from strips were the same as described above.

#### **Greenhouse Experiment**

Eight surface soils (0-15 cm) representing 4 orders (Mollisol, Vertisol, Oxisol, and Ultisol) and a wide range in P sorption capacity were included in this study. Soils were air-dried and ground to pass a 4-mm sieve. A representative sample of each soil was ground to pass a 0.84-mm sieve, and analyzed for pH at 1:1 soil/water ratio (w/w), organic carbon with the Walkley and Black method (Nelson and Sommers, 1982), clay content by the method of Gee and Bauder (1986), and calcium carbonates by the method of Nelson (1982) (Table 1).

Based on P sorption capacity, the soils were divided into three groups: low, medium, and high (Table 2). There were 4 treatments (Table 2) for each soil with three replicates arranged in a Randomized Complete Block design.

Acidic soils were limed to pH 6.5 by adding  $CaCO_3$  powder according to the single buffer solution method (Adams and Evans, 1962). After liming, the soils were brought to field capacity and incubated for 4 weeks in plastic bags. Five-kg (dry basis) subsamples of each soil were weighed onto clean brown paper sheets. The treatment amount of P fertilizer ( $Ca(H_2PO_4)_2 \cdot H_2O$ ) was thoroughly mixed with each sample. The soil samples were then placed into

**TABLE 1.** Selected properties of the soils.

Series	Subgroup	pH (H <sub>2</sub> O) (1:1, W/W)	Organic C	Clay (<2\(\mu\m)	CaCO <sub>3</sub>
			0.7042000000000	g kg <sup>-1</sup>	
Honouliuli	Typic Chromustert	7.26	16.6	580.0	3.2
Lualualei	Typic Chromustert	7.65	4.1	788.4	16.2
Nohili	Cumulic Haplaquoll	7.44	17.6	806.6	65.8
Paaloa	Rhodic Kandiudult	5.05	40.0	740.8	-†
Wahiawa	Rhodic Eutrustox	5.05	35.8	895.0	<u>.</u>
Kapaa	Anionic Acrudox	4.92	45.6	525.1	-
Leilehua	Ustic Kanhaplohumult	4.66	44.0	862.9	-
Mahana	Rhodic Haplustox	4.31	36.7	486.3	-

<sup>†</sup> Negligible.

**TABLE 2.** Phosphorus application rates for soils of greenhouse experiment.

	P applied to achieve target solution P levels mg P L <sup>-1</sup>			
Series	Control	0.2	0.5	1.0†
	mg P kg <sup>-1</sup>			
Low P sorption				
Honouliuli, Lualualei, and Nohili	0	100	250	500
Medium P sorption				
Paaloa, and Wahiawa	0	500	650	800
High P sorption				
Kapaa, Leilehua, Mahana	0	1400	1800	2200
- ·				

<sup>†</sup>Determined by the Fox and Kamprath method (1970).

plastic pots (18 cm in diameter, 30 cm high), and watered with deionized water to field capacity and incubated in the greenhouse for 4 weeks. Soils were then analyzed for Pi P, Olsen P (Olsen and Sommers, 1982) and Mehlich-1 P (Mehlich, 1953) before seeding. Basal nutrients, including 50 mg N kg<sup>-1</sup> as urea, 60 mg K kg<sup>-1</sup> as KCl, 25 mg kg<sup>-1</sup> Mg as MgSO<sub>4</sub>, and 5 mg kg<sup>-1</sup> Zn as ZnSO<sub>4</sub>, were added into each pot and mixed.

In each pot, 12 pregerminated corn seeds (Hybrid X304CF15, courtesy of Pioneer Hi-Bred International, Inc., Johnston, Iowa) were placed about 1 to 2 cm below the soil surface. At 3 days after emergence, each pot was thinned to 10 seedlings. Two additional doses of N at 50 mg/kg were applied at 10 and 20 days after emergence. Plants were allowed to grow for 4 weeks. The pots were watered daily and moisture was maintained near field capacity throughout the growth period. At harvest, plants were cut at the soil surface, washed with deionized water, dried at 70°C, and ground to pass a 0.5-mm sieve. Phosphorus in tissue was determined by ashing 0.05-0.1 g sample and dissolving the ash in 1.0 ml 1M HCl, which was then diluted to 10 ml. Solution P was determined by the method of Murphy and Riley (1962). A 15-g soil sample was taken from each pot, dried and ground to pass a 20-mesh sieve. Olsen P, Mehlich 1 P, and Pi P were determined.

#### **RESULTS AND DISCUSSION**

#### Characterization of Iron on the Pi Strips

X-ray diffraction (XRD) technique was used in this study to reveal the nature of the Fe material on the Pi strips. Strips prepared with both Whatman No. 50 and 541 had identical XRD patterns.

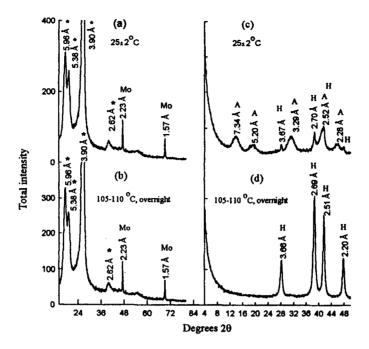


FIGURE 1. X-ray diffractograms of Fe materials on Pi strips Whatman No. 50, washed) (a and b) and from mixing FeCl<sub>3</sub> and NH<sub>4</sub>OH solutions (c and d). Peaks with an \* were due to the cellulose of the filter paper. H = hematite  $(\alpha\text{-Fe}_2\text{O}_3)$ , A = akaganeite  $(\gamma\text{-FeOOH})$ , and Mo = peaks from the molybdenum sample plate.

Figure 1a and 1b are air-dried and heated XRD patterns, respectively, of the Pi strips prepared with Whatman No. 50 filter paper. It appeared that no crystalline Fe compounds were present on the air-dried Pi strips (Figure 1a). The distinct peaks were due to the cellulose of the filter paper and the molybdenum sample plate. We expected that heating the Pi strips might promote more crystallization and concentrate crystalline Fe on the Pi strips. However, no crystalline phase was observed on strips even after they were heated overnight at 105-110°C (Figure 1b).

The XRD patterns of Fe precipitate obtained by mixing FeCl<sub>3</sub> and NH<sub>4</sub>OH for about 1 minute showed crystalline Fe phases. The air-dried precipitate contained hematite (α-Fe<sub>2</sub>O<sub>3</sub>) and akaganeite (β-FeOOH) (Figure 1c). When the precipitates were heated at 105-110°C overnight, all of the Fe was converted into hematite (Figure 1d).

The reactions for preparing Pi strips are different from direct mixing of FeCl<sub>3</sub> and NH<sub>4</sub>OH, because they occur on filter papers. The filter paper matrix may retard the nucleation and crystal growth of the reaction products. However, some similarities are expected. In both cases, rapid precipitation occurs. It is expected that amorphous or meta-crystalline Fe hydroxides may be the immediate products (McLaughlin et al., 1981; Schwertmann and Taylor, 1989). Such Fe hydroxides may partially dehydrate to form Fe oxides if they are isolated from water (Schwertmann and Taylor, 1989). However, when Fe coated on the filter paper is widely dispersed on the fiber matrix of the paper, extensive crystallization of Fe may not occur. Crystalline Fe on the strips may be so dilute that no phase peaks can be identified on the XRD patterns (Figure 1 a, b).

We believe that Pi strips at room temperatures may contain both hydrous Fe hydroxides and oxides in both crystalline and amorphous forms. Crystalline Fe oxides such as hematite are weaker P sorbers compared with amorphous Fe oxides and hydroxides (McLaughlin et al., 1981). It has been reported that drying of amorphous Fe at 80°C greatly decreases its P sorption capacity (McLaughlin et al., 1981). Phosphorus sorption capacity of strips should decrease after heating if the same process occurs when strips are heated as occurs with direct reaction products. Such P sorption capacity decrease of the Pi strips by heating was reported in a study

by Lin et al. (1991), in which they found that P sorbed per strip was much lowered by prolonged drying at 70°C. This is indirect evidence of the presence of amorphous Fe on the Pi strips.

Because Fe on the Pi strips may undergo phase changes, both the composition and P sorption capacity of Fe on the strips vary depending on the conditions under which they are prepared and stored. The uncertainty of the Fe components on the strips is one of the difficulties of the Pi methodology.

#### P Sorption Capacity and Extraction Error of Pi Strips

Phosphorus sorption capacity and extraction error depend on filter paper, FeCl<sub>3</sub> concentration, contact time between FeCl<sub>3</sub> and the filter paper, reaction time between NH<sub>4</sub>OH and the FeCl<sub>3</sub>-treated paper (Lin et al., 1991), and whether or not the strips are washed with deionized water after they are treated with NH<sub>4</sub>OH (Bramley and Roe, 1993).

Various filter papers have been used for preparing Pi strips. Whatman No. 50 and 541 were used in this experiment because of their wide availability and because they were used in most Pi studies.

Lin et al. (1991) found that contact times between FeCl<sub>3</sub> solution and filter paper longer than 2.3 h did not make any difference in Fe content on the strips. Thus a 2-h contact time was used in this study.

Conversion of FeCl<sub>3</sub> to high P-affinity Fe was realized by reacting FeCl<sub>3</sub>-treated paper with aqueous NH<sub>4</sub>OH. Early procedures suggested drawing the paper swiftly through NH<sub>4</sub>OH solution. More recent studies have recommended that FeCl<sub>3</sub> treated paper be immersed in NH<sub>4</sub>OH for 1 min. for even deposition of Fe material on the papers (Sharpley et al., 1994). FeCl<sub>3</sub>-treated paper with NH<sub>4</sub>OH in aqueous solution produces Fe compounds which are loosely bound

TABLE 3.	Mean Fe content of strips as affected by type of filter paper
	and washing.

Paper type	Post-reaction treatment	Fe (μmol/strip)†	
Whatman No. 50	washed	77.00 c‡	
Whatman No. 50	unwashed	82.73 b	
Whatman No. 541	washed	90.96 a	
Whatman No. 541	unwashed	93.83 a	

<sup>†</sup> Mean of 10 determinations.

to the paper. Therefore, it is necessary to wash the Pi strips for consistent results (Bramley and Roe, 1993).

To further minimize inconsistencies in strip preparation, we found it best to air-dry FeCl<sub>3</sub>-treated papers overnight before they are treated with aqueous NH<sub>4</sub>OH. Moist FeCl<sub>3</sub>-treated filter papers contaminate the NH<sub>4</sub>OH solution with Fe materials, and uneven Fe deposition on the strips may occur.

The amount of Fe on the strips varied with paper and preparation (Table 3). Strips from Whatman No. 50 contained significantly less Fe than those from Whatman No. 541. Iron contents were more consistent on washed strips than on unwashed strips as indicated by the smaller differences in standard deviation (Table 3). Within the same paper type, washing NH<sub>4</sub>OH-treated paper significantly reduced Fe content on Pi strips prepared from Whatman No. 50, but not on strips from Whatman No. 541. Whatman No. 50 is a thicker paper than Whatman No. 541, and we speculate that the former has finer

<sup>‡</sup> Means followed by the same letter are not significantly different at 1% level, LSD<sub>.01</sub>=4.48  $\mu$ mol/strip.

pores and less pore space than does the latter. It may also be possible that strips from Whatman No. 541 have more interior pore space than strips from Whatman No. 50. Therefore, strips from Whatman No. 541 can hold more Fe than strips from Whatman No. 50 for the same size strips, and less Fe is affected by washing.

Iron content and the response to washing of different strips influence both P sorption capacity and extraction error of the strips. Figure 2 shows the P extraction capacity of strips in a series of P solutions. All strips showed a linear extraction range below 300 µg added P. However, when larger amounts of P were added, Pi strips could not fully recover the added P and a saturation range exists. Phosphorus sorption maxima increased with increasing Fe contents on the Pi strips (Figure 2). The saturation range is practically insignificant because such high P levels rarely occur in soil solutions. If such high P levels exist, the Pi will be unsuitable unless a smaller amount of soil is used for extraction.

Within the linear extraction range (0-300 µg P/strip), P recovered was not affected by the differences in Fe content of the four types of strips, and accounted for more than 96% of the added P (Figure 2). This suggested that within this range, one Pi strip is sufficient to deplete P in soil solutions with very high P levels. Current Pi tests use 1 g of soil in 40 ml of electrolyte solution. Even in soils with extremely high P levels, one strip should be enough for successful P extraction.

Although both filter papers are hardened papers, shaking strips in soil suspension resulted in fine fibers and loosely bound Fe coming off the strips (Lin et al., 1991, Bramley and Roe, 1993). Use of a nylon sheath to entrap the strips was recommended (Menon et al,

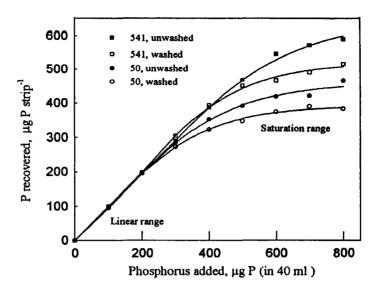


FIGURE 2. Phosphorus sorption capacities of Pi strips prepared with two filter papers and two procedures. Numbers "50" and "541" refer to Whatman No. 50 and 541 filter papers, respectively.

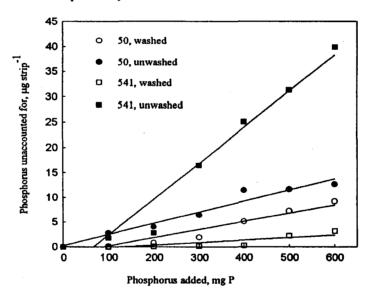


FIGURE 3. P unaccounted for as affected by Pi strip preparation. Numbers "50" and "541" refer to Pi strips prepared with Whatman No. 50 and 541 filter papers, respectively.

1989a); however, such a measure did not completely eliminate the problem. Phosphorus bound with Fe detached from strips is neither recovered by the strips nor is measured in solution. Such unaccounted-for P is referred to as extraction error.

As was shown in Figure 3, extraction error was greater with unwashed strips prepared from both filter papers. Washed strips from Whatman No. 541 showed the smallest extraction error. However, less soil contamination of the strips by fine soil particles during extraction was found for strips from Whatman No. 50 paper than strips prepared with Whatman No. 541 paper (Myers et al., 1995). In this study, soil contamination was minimized by directing a jet of deionized water towards the strips to wash the strips free of visual particulate matter. Both papers appear to be appropriate Fe carriers because strips from both papers endured vigorous shaking and washing without being torn apart when nylon bags were used to entrap the strips during shaking. Therefore, washed strips prepared from both Whatman No. 50 and 541 were used for the remainder of this study.

### Greenhouse Evaluation of the Pi, Olsen, and Mehlich 1 Methods

Relationships Between Pi Phosphorus and Extractable Phosphorus by Olsen and Mehlich 1. A useful first step for evaluating a new soil P test is to compare extractable P by the new method with P extracted by a relatively well established test or a test that has been recommended for the soils of interest. Although a good correlation does not necessarily validate the new method (Fixen and Grove, 1990; Sharpley, 1991), a poor correlation between a new method and a

relatively well established test is strong evidence to reject the new method (Fixen and Grove, 1990).

Extractable P by Mehlich 1, Olsen, and Pi in soil samples of the first three crops of the greenhouse experiment were presented in Figure 4. All tests were performed immediately prior to planting. Both Pi P and Mehlich 1 P were plotted against Olsen P because Olsen method is more adaptable to different soils than Mehlich 1 (Fixen and Grove, 1990). Although both Pi P and Mehlich 1 P were highly correlated with Olsen P (significant at 0.1% level), Pi P (from both paper types) was more closely related to Olsen P, as indicated by their respective coefficients of determination (r²) (Figure 4). The Mehlich 1 method extracted little P from highly calcareous soils. With Nohili soil, Mehlich 1 responded poorly to additional P increases (data points close to X axis of Figure 4), because the acidity in the extractant was either partially or completely neutralized during extraction (0.75% of carbonates neutralizes 50% of the acidity in the Mehlich 1 extractant).

The Pi method may be more applicable to diverse soils because it does not seem to have the disadvantage of reduced extraction efficiency on calcareous soils observed with the Mehlich 1 method. The Pi method appears to be more suitable in soils to which rock phosphate was added (Menon et al., 1989a, 1989b), while the Olsen method has worked well on some soils (Yost et al., 1982), but was ineffective on others (Menon et al., 1989a, 1989b).

Extractable Phosphorus by Mehlich 1, Olsen, and Pi and Plant Phosphorus Uptake. The response of extractable P by a new P test to P applications, and to plant response such as growth, and plant P

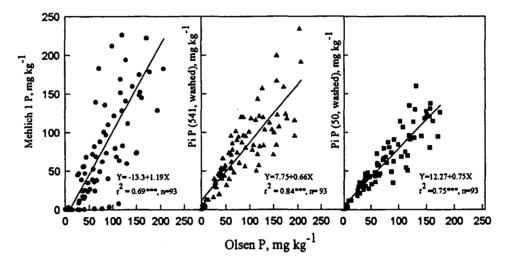


FIGURE 4. Relationships between Olsen-P and extractable-P by the Mehlich 1 method and Pi strips. Soil samples were collected before planting each of the first three crops of a greenhouse experiment.

uptake are usually considered the critical, most important criteria for assessing new P test methods.

The greenhouse experiment was initially designed to test the differences in residual available P among various soils. The two highest P addition rates were sufficiently high so that available soil P can sustain growth of several crops. As was expected, the plant growth response curve for each of the first several crops of each soil showed a plateau. We believe that when extractants are evaluated by plant growth response, the most meaningful comparisons should be made when plant growth response to P additions is linear, although quadratic response curves can provide limited information. Therefore, the relationship between corn biomass and extractable P was not used as an evaluating criterion. Correlation coefficients between P uptake of corn and extractable P of the first 3 harvests are summarized in Table 4.

Honouliuli, Lualualei, and Nohili are soils with pH greater than 7.0, and with low P sorption capacities. The Mehlich 1 P correlated well with P uptake by each crop in Honouliuli and Lualualei, but not as well in Nohili. For the first crop of Nohili, Mehlich 1 P correlated well with P uptake. But as cropping continued, correlations between extractable P and P uptake became insignificant at the 5% level (Table 4). Nohili had the highest carbonate content (65.8 g kg<sup>-1</sup>). For the first crop of Nohili, soil solution P was high. Mehlich 1 extractant could detect the differences in solution P corresponding to different added P levels. However, when soil solution P was largely removed, Mehlich 1 was not strong enough to differentiate different P levels in soils. Lualualei soil had 16.2 g kg<sup>-1</sup> total carbonates. Mehlich 1 P was considerably lower compared with non-carbonaceous soil

TABLE 4. Correlation coefficients between phosphorus uptake (mg P/pot) of corn and extractable phosphorus by different methods†.

Soil	Crop	Olsen	Mehlich 1	Pi-541, washed	Pi-50, washed
Honouliuli	1	0.961	0.963	0.966	0.980
Honounun	2	0.985	0.959	0.980	0.950
	3	0.991	0.991	0.972	0.995
Lualualei	1	0.981	0.935	0.954	0.990
Dualuaici	2	0.870	0.945	0.863	0.936
	3	0.951	0.906	0.951	0.971
Nohili	1	0.931	0.827	0.903	0.892
1401111	2	0.866	0.622*1	0.868	0.889
	3	0.896	0.553ns	0.895	0.849
Paaloa	1	0.830	0.934	0.917	0.952
1 aai0a	2	0.953	0.971	0.965	0.937
	3	0.933	0.925	0.861	0.933
Wahiawa	1	0.989	0.939	0.942	0.980
w ашаwа	2	0.983	0.983	0.978	0.991
	3	0.983	0.993	0.974	0.983
Kapaa	1	0.946	0.942	0.954	0.984
Kapaa	2	0.940	0.942	0.934	0.996
	3	0.984	0.946	0.980	0.930
Leilehua	3 1		0.936	0.937	0.920
Lenenua	_	0.977 0.972	0.890	0.910	0.906
	2 3			0.900	0.920
Mahana	3 1	0.914	0.861	0.838	0.934
Mahana	_	0.949	0.966		
	2	0.953	0.902	0.901	0.977
	3	0.994	0.920	0.986	0.983

<sup>†</sup> Twelve observations for each soil in each crop.

<sup>‡</sup> All coefficients were significant at 0.1% level unless otherwise indicated.

<sup>\*</sup> indicates significant at 5% level, and ns=not significant at 5% level.

samples with similar extractable P levels by the Olsen method. But Mehlich 1 P correlated well with P uptake in Lualualei. Honouliuli has the least amount of carbonates among the three soils and Mehlich 1 was a suitable extractant. In these 3 soils, both Olsen P and Pi P correlated equally well with P uptake (Table 4). Therefore, these two methods can be used for soils with pH greater than 7.0, and do not appear sensitive to the presence of soil carbonates.

Paaloa and Wahiawa are highly weathered soils with low pH and medium P sorption capacity. Kapaa, Leilehua, and Mahana are highly weathered soils with high P sorption capacity. Mehlich 1 is recommended for such soils, and the Olsen method performs reasonably well on acid soils (Smyth and Sanchez, 1980). In this study, all three tests predicted P uptake well in these soils (Table 4). It appears that all three methods may be used for such soils.

Although correlation coefficients between extractable P and plant P uptake are useful in preliminary evaluations of extractants, such coefficients are by no means conclusive. A significant correlation between two sets of data will result whenever there is a similar trend. When correlations are used in evaluating P extractants, some critical components for legitimate evaluations, such as extractant precision, accuracy, and data variations are often not reflected in such correlations. An alternative method for evaluating test methods for measuring available P have been proposed by some workers (Cai et al., 1996); however, extensive efforts are needed for validating their proposed approach.

<u>Differences in Measuring Available Phosphorus Between Pi</u> and Chemical Extractants. As attempts are made to increase P fertilization accuracy, we seek quantitative estimates of the available soil P pools in addition to numbers that correlate well. The exhaustion study is one way to directly measure plant-usable P pools in ways not possible by correlations alone. Studies have suggested that the mode of P extraction of the Pi is fundamentally different from that of chemical extractants such as the Mehlich 1 and Olsen methods (van der Zee et al., 1987; Menon et al., 1989a). Therefore, in this study an attempt was made to compare the abilities of Mehlich 1, Olsen, and the Pi to predict available P pools in soils as measured by an exhaustion method.

Accumulated plant available P as estimated by P removal by eight consecutive crops of corn at various P levels for 8 soils was correlated with extractable P by the Mehlich 1, Olsen, and the Pi methods prior to planting of the first crop. Correlation coefficients between extractable P by all three methods and accumulated plant available P were significant at the 0.1% level (Figure 5). Among the three methods, the Pi P predicted plant available P best, followed by Olsen and Mehlich 1. Between the two types of Pi strips, Pi-50 was slightly better in predicting plant available P than was Pi-541. However, as the results in Figure 5 suggest, extractable P by any method alone cannot explain all the variation of plant available P. Even Pi-50 accounted for only about 61% of the variation. Pi strips remove only loosely bound soil P (Sharpley, 1991). Phosphorus associated with amorphous aluminum, Fe and calcium compounds is not readily extracted by Pi strips (Sharpley, 1991; van der Zee et al., 1987). The mechanism of Pi extraction may be more analogous to P sorption by plants compared with chemical extractants. Similar to chemical extractants, Pi strips still do not accurately measure plant P removal from soils.

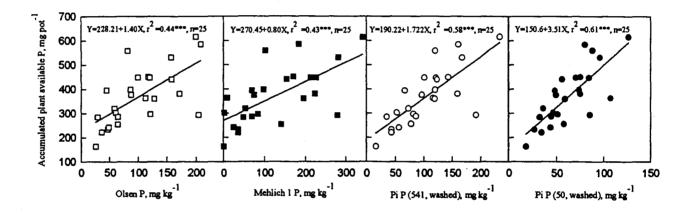


FIGURE 5. Relationships between accumulated plant-available-P and extractable-P by different methods. Accumulated plant-available-P estimated as total-P removal by 8 consecutive crops.

#### CONCLUSIONS

This study showed that Pi strips at room temperatures may contain both hydrous Fe hydroxides and oxides in both crystalline and amorphous forms. Pi strips prepared from different types of filter paper contained different amounts of Fe, and the P adsorption maxima of strips were proportional to the amounts of Fe. Strips prepared from Whatman No. 50 paper had significantly less Fe per strip than strips from Whatman No. 541. Washing the strips with deionized water removed the loosely bound Fe from the strips and reduced P extraction error. Thus, washing appears to be necessary for reducing error of the Pi method.

Pi strips from both Whatman No. 50 and 541 showed a linear extraction range (0-300 $\mu$ g P/strip) and a saturation range (>300  $\mu$ g P/strip). For most soils, one strip is adequate to estimate the available P usually present in 1 g of soil.

Based on the results of this study and information in the literature, we recommend the following Pi strip preparation procedure:

Soak Whatman No. 50 or 541 filter paper in 0.4M FeCl<sub>3</sub> solution for 2 h and air-dry overnight. Then immerse the paper in 2.7M NH<sub>4</sub>OH for 1 min. Air dry overnight and wash NH<sub>4</sub>OH-treated papers with deionized water. Air dry paper and cut them into 2 cm x 10 cm strips. Store strips at room temperature in closed containers.

Both Pi P and Mehlich 1 P were well correlated with Olsen P for a range of soils. Pi P was more closely related to Olsen P than Mehlich 1 P. Unlike the Mehlich 1 method, both the Pi and the Olsen method were insensitive to soil carbonates. Therefore, Pi and Olsen appear to be more adaptable to diverse soils. Both Pi P and

Olsen P were significantly correlated with plant P uptake in all soils, indicating the general applicability of Pi P. No obvious advantage was found with Pi compared with Olsen. However, Pi was more suitable in soils to which rock phosphate had been applied (Menon et al., 1989a, 1989b) than the Olsen method.

Extractable P by all three methods was highly correlated with accumulated plant available P estimated by eight sequential crops in the greenhouse. However, none of the three methods could explain all the variation in plant available P. Although the mechanism of Pi extraction is more analogous to P uptake by plants than chemical extractions, Pi P is still not an accurate measure of plant-available soil P.

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